भारतीय मानक
Indian Standard

IS 538 : 2020

फिनोल (कार्बोलिक) अम्ल — विशिष्टि

(तीसरा पुनरीक्षण)

Phenol (Carbolic Acid) — Specification

(Third Revision)

ICS 71.080.90

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

July 2020 **Price Group 6**

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

FORWARD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards after the draft finalized by Organic Chemicals, Alcohols and Allied Products Sectional Committee had been approved by the Petroleum Coal and Related Products Division Council

Phenol is wide used for polymerization with formaldehyde to produce phenolic resins commonly used for laminate industry, Bakelite products, plastic industries, chemical intermediate, motor lubricants, pharmaceuticals, astringents and derivative of phenol is used as an antiseptic, germicide and disinfectant. It is also used in the manufacture of certain dyes and picric acid.

This standard was first published in 1955 and subsequently revised in 1968 and 2000. In the first revision, requirement for boiling point was omitted and use of a dehydrated sample in the determination of crystallization point was stipulated. However, the committee responsible for preparation of this standard had decided to revise it again in accordance with the development in and growing demand by the laminated and petrochemical industry. In the second revision of the standard two grades of the material were introduced on the basis of assay and crystallization point. The grades were also meant for specific use like pharmaceuticals and for general industrial purposes.

Further, the third revision has been undertakenby the technical committee to reduce the impact of impurities by increasing the purity of phenol and also to meet the compliance of environment and human society as well as pharmaceutical need. To achieve this, GC analysis has been revised to FID capillary GC system to detect trace impurities instead of conventional and packed column GC methods.

The composition of the Committee, responsible for the formulation of this standard is given at Annex F.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PHENOL (CARBOLIC ACID) — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for high purity phenol (carbolic acid) and toxic impurities.

2 REFERENCES

The following standards contain provisions which through, reference in this text constitute the provisions of the standards. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

IS No./Other Publications	*Title*
82 : 1973	Methods of sampling and test for thinners and solvents for paints (*first revision*)
265 : 1993	Specification for hydrochloric acid (*fourth revision*)
1070 : 1992	Reagent grade water or Millipore water (*third revision*)
1260 (Part 1) : 1973	Pictorial marking for handling and labeling Of goods: Part 1 Dangerous goods (*first revision*)
2362 : 1973	Determination of water by the Karl Fischer method or auto Karl Fischer traitor (*first revision*)
4905 : 2015/ ISO 24153 : 2009	Random sampling and randomization procedures (*first revision*)
5296 : 1995	Chloroform, pure and technical (*second revision*)
1448 (Part 40) : 2015/ISO 3733 : 1999	Methods of test for petroleum and its products: Part 40 Petroleum products and bituminous materials — Determination of water — Distillation method
6270 : 1971	Code of safety for phenol
8768 : 2000	Method of measurement of colour in liquid chemical products platinum — Cobalt scale (*second revision*)
ASTM D1686-19	Standard Test Method for Color of Solid Aromatic Hydrocarbons and Related Materials in the Molten State (Platinum-Cobalt Scale)
ASTM D5386-16	Standard Test Method for Color of Liquids Using Tristimulus Colorimetry
ASTM D1209 05 (2019)	Standard Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)

3 GRADES

The material shall be of two grades, namely:

a) Grade A; and

b) Grade B.

4 REQUIREMENTS

4.1 Description

The material shall be colourless to light pink liquid and free from suspended matter at 41°C. Phenol may also be supplied as hydrated phenol containing a specified quantity of water (4 to 10 percent by mass) as agreed to between the purchaser and the supplier. In such cases the phenol content of the hydrated phenol shall be determined either by gas chromatography and water content by Karl Fischer or by brominating method and water content by IS 1448 (Part 40) using 2 or 10 ml receiver depending on water content. However, the brominating method does not give impurity profile of phenol.

4.2 Solubility in Water

When 10 g of the material is mixed with 120 ml of distilled or demineralized water, the solution shall be complete and clear, and shall remain so after standing for 30 min at 27°C.

4.3 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods given in last column of Table 1.

Table 1 Requirement for Phenol (Carbolic Acid)

(*Clauses* 4.3 *and* 7.1)

Sl. No.	Characteristic	Requirement		Method of Test, Ref in Annex
		Grade A	Grade B	
(1)	(2)	(3)	(4)	(5)
i)	Crystallization point, °C, *Min*	40.3	39.5	**A**
ii)	Moisture, present by mass, *Max*	0.1	0.1	**B**
iii)	Residue on evaporation by mass, *Max*	0.05	0.05	**C**
iv)	Purity percent by mass	99.8 *Min*	98.5 to 99.8	**D-3**
vi)	Colour and APHA/HU	As agreed to between the purchaser and the supplier		IS 8768 or ASTM D 1686 or ASTM D 5386 or ASTM D 1209

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in amber colored or dark blue glass bottles fitted with ground glass stopper or as agreed to between the purchaser and the supplier. Small size packings are being used for marketing development, customer sample, laboratory analytical grade etc. Phenol being hazardous material, refer in IS 6270.

5.2 Marking

5.2.1 Each container shall be securely closed and shall bear legibly and indelibly the following information

a) Name and grade of the material;

b) Lot or batch number and date of manufacture;

c) Tare, gross and net mass; and

d) Indication of the source of manufacture.

5.2.2 Each container shall also be marked with Fig. 1 of IS 1260 (Part 1) along with the following information printed in the space provided.

POISON

'PROTECT FROM LIGHT AND MOISTURE'

5.2.3 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.'

6 SAMPLING

The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Annex E.

7 TEST METHODS

7.1 Test shall be conducted as prescribed in last column of Table 1.

7.2 Quality of Reagents

Unless otherwise specified, pure chemicals and distilled water (*see* IS 1070) shall be employed in the tests

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities. Which affect the results of analysis.

ANNEX A

[*Table* 1 *Sl No.* (i)]

DETERMINATION OF CRYSTALLIZATION POINT

A-1 APPARATUS

A-1.1 Crystallization-point Apparatus

The apparatus is illustrated in Fig. 1. A glass tube 150 x 25 mm nominal size is placed inside a 175 x 38 mm test tube. The latter tube is flanged so that it may be supported centrally by a metal cover plate in a 1 000 ml tall-form beaker filled with water to within 20 mm of the top. The wider tube is weighed with lead shots or similar material and the inner tube is closed by means of a cork which carries a glass stirrer and through its centre a standard thermometer. The stirrer has a loop of outside diameter of about 18 mm, surround the thermometer. The thermometer is so fixed in the cork that the bottom of the bulb is about 15 mm from the bottom of the inner tube. A thermometer for the water-bath passes through a hole in the cover plate and is held by a rubber ring.

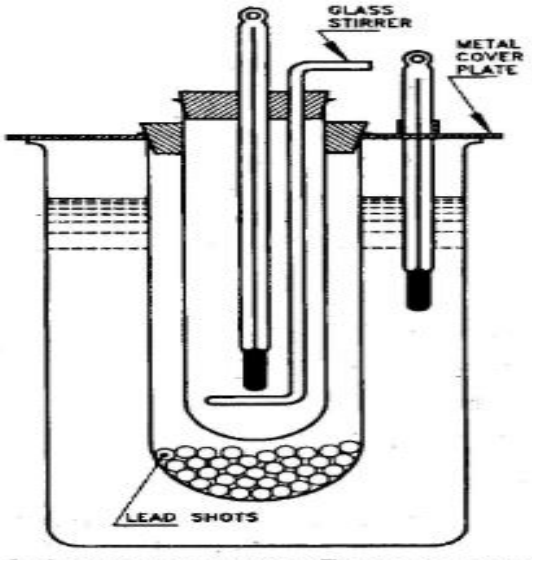

FIG. 1 APPARATUS FOR THE DETERMINATION OF CRYSTALLIZING PONT

A-1.2 Thermometer

Conforming to the following essential requirements:

Range	15 to 45 °C
Graduation	0.1 °C
Longer lines, at each	0.5 and 1°C
Fully figured, at each	2 °C
Overall length, *Max*	430 mm
Lengthofmainscale, *Min*	260 mm

NOTES:

1 Any thermometer of similar requirements and accuracy may be used.

2 The thermometer shall bear a certification from the national or international accredited certified body or NABL certified laboratory or equivalent.

A-1.3 Water-Bath

A-1.4 Measuring Cylinders

50 ml and 10 ml capacities.

A-2 PROCEDURE

A-2.1 Determination of Crystallization Point

Remove the inner tube from the crystallization point apparatus and directly pour about 20 ml of sample (in liquid form) into it. Insert the cork carrying the thermometer and stirrer. If the sample has commenced to crystallize. Heat gently until it becomes liquid and then cool rapidly to determine the approximate crystallizing point. Warm the tube in the water-bath at about 5°C above this point, so that the crystals melt, except for a trace necessary for seeding. Replace the inner tube in its jacket with the water in the apparatus maintained at a temperature between 6 and 8°C below the expected crystallizing point. Stir the sample gently and continuously and record thermometer readings at 30 s intervals. The crystallizing point corresponds to the highest of the first five consecutive readings during which the temperature remains constant within 0.1°C.

If super cooling occurs, as shown by a rise in temperature, observe the constant temperature after the rise. A temperature rise of 1°C is the maximum allowable. If five consecutive readings commencing with the point at which the maximum temperature is first attend plot the complete cooling curve of temperature against time and draw a straight line to lie events between the first and second and between the fifth and sixth points mentioned above extend this line to meet the section of cooling curve before the temperature rise. Report the temperature corresponding to the point of intersection as the crystallizing point.

ANNEX B

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF MOISTURE

B-1 PROCEDURE

Determine the moisture content in phenol by Karl Fischer method as prescribed in IS 2362.

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF RESIDUE ON EVAPORATION

C-1 APPARATUS

C-1.1 U – Tube Stopperes

With limbs about 125 x 15 mm. Each limb is provided with a side tube as shown in Fig. 2. The stoppers shall not be greased.

C-1.2 Air Bath

A copper air bath about 150 x 150 x 150 mm.

C-1.3 Thermometer

Conforming to the following essential requirements:

Range	50 to 210°C
Graduation	0.5°C
Longer lines, at each	1 and 5°C
Fully figured, at each	10°C
Overall length, *Max*	430 mm
Length of main, scale	240 mm
Error, *Max*	+0.8°C

NOTES:

1 Any thermometer of similar requirements and accuracy may be used.

2 The thermometer shall bear a certification from the national or international accredited certified body or NABL certified laboratory or equivalent.

C-1.4 Calcium Chloride Tube

C-1.5 Conical Safety Flask

C-1.6 Suction Pump

C-2 PROCEDUREC

C-2.1 Assemble the apparatus as shown in Fig. 2 without the U-tube. Heat the air bath 150 ± 5 °C and maintain it at this temperature. Connect the U-tube and draw a gentle stream of air through it for approximately half an hour. Remove the U-tube. Connect it by a side arm to the calcium chloride tube and close the other side arm to the atmosphere. When cool, remove the calcium chloride tube and weigh the u-tube to within 0.0001 g with both stoppers closed.

C-2.2 Weigh accurately about 10 g of the sample into the U-tube, at the lowest temperature for complete liquidity. Reconnect the U-tube to the train and make sure that the bottom of the thermometer bulb is level with the bottom of U-tube.

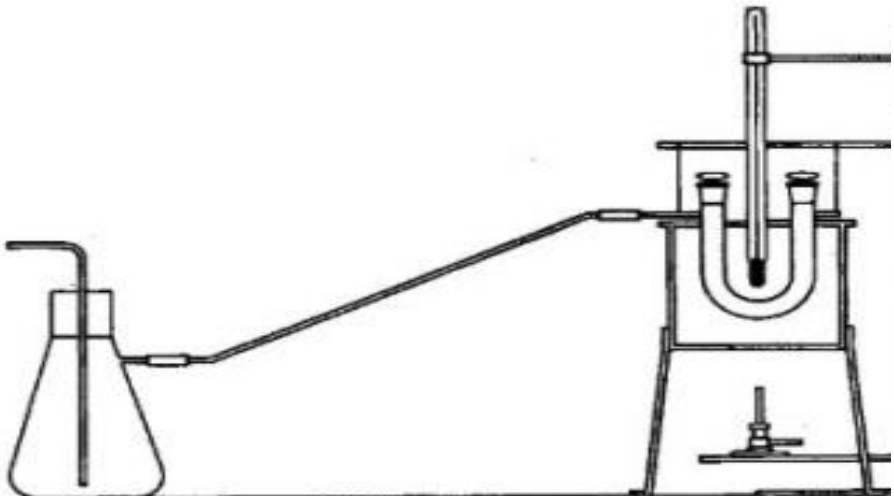

FIG. 2 APPARATUS FOR THE DETERMINATIOB OF RESIDUE ON EVAPORATION

C-2.3 Draw a gentle stream of air through the apparatus and maintain the air-bath at 150 ± 5 °C until evaporation is complete. Disconnect the U-tube, fit the calcium chloride tube to one side arm and close the other to the atmosphere. When the tube is cool, weigh it as before. Repeat the heating, cooling and weighing until a constant mass is obtained

NOTE — Clause **8** of IS 82 may also be used for residue on evaporation test. Phenol is a high boiling liquid, hence, hotplate should be used (control heating facility) instead of water bath.

C-3 CALCULATION

$$\text{Residue on evaporation, percent by mass} = \frac{M^1}{M^2} \times 100$$

Where,

M_1 = mass in g, of the residue left; and

M_2 = mass in g, of the sample taken for the test.

ANNEX D

[*Table* 1*, Sl No.* (iv)]

DETERMINATION OF ASSAY

D-1 GENERAL

Two methods have been specified for determination of assay of phenol:

a) Method A is bromide-bromate volumetric method or conventional method, and

b) Method B is the gas chromatographic method.

In case of dispute of method B shall be the referee method.

D-2 BROMIDE-BROMATE SOLUTION METHOD (METHOD A)

D-2.1 Outline of the Method

The method is based on the reaction between phenol and bromine from standard bromide-bromate solution. Other phenols and unsaturated compounds react with bromine and if these are present they will invalidate the results.

D-2.2 Reagents

D-2.2.1 *Chloroform (see IS 5296)*

D-2.2.2 *Concentrated Hydrochloric Acid, (see IS 265).*

D-2.2.3 *Potassium Iodide Solution, 20 Percent (m/v)*

Dissolve 20 g of potassium iodide in 100 ml of water

D-2.2.4 *Standard Bromide-bromate Solution, 0.1 N*

Dissolve 10.2 g of potassium bromide and 2.784 g of potassium bromate in water and dilute to 1 000 ml in a volumetric flask.

D-2.2.5 *Standard Solution Thiosulphate Solution, 0.1 N*

Dissolve 25 g of sodium thiosulphate in water and dilute to 1 000 ml. It is essential to find out the factor of the sodium thiosulphate solution immediately before use, as follows:

Pipette 25 ml of the bromide-bromate solution into the iodine flask. Add 5 ml of the potassium iodide solution, followed by 5 ml of hydrochloric acid. Stopper immediately and shake thoroughly. Titrate the liberated iodine with the sodium thiosulphate solution until the contents of the flask become only faintly yellow, add a few drops of starch solution and continue the titration until the last traces of the blue colour have disappeared.

The factor ‘f’ of the sodium thiosulphate solution is 25/v, where is the volume (ml) of soium thiosulphate solution used.

D-2.2.6 *Starch Solution*

Freshly prepared.

D-2.3 Apparatus

D-2.3.1 *Iodine Flask*

500 ml capacity.

D-2.3.2 *Measuring Cylinders*

One of 100 ml and three of 25 ml capacities.

D-2.3.3 *Pipettes*

50 ml and 25 ml capacities.

D-2.3.4 *Burette*

50 ml capacity

D-2.4 Procedure

Weigh accurately about 2.5 g of the sample and dissolve it in water, dilute to 1 000 ml with water in a volumetric flask. Pipette 25 ml of this solution into the iodine flask and add 50 ml of the bromide-bromate solution, followed by 5 ml of hydrochloric acid. Stopper immediately and seal by running a little of the potassium iodide solution into the annular space between the stopper and the funnel-shaped neck of the flask. Mix the contents of the flask by occasional swirling during half an hour and then leave to stand for a further period of 15 min. Gradually add 15 ml of potassium iodide solution to the contents of the flask by pouring the solution into the annular space on the neck and gently easing the stopper. Shake the flask thoroughly and titrate the contents with the standards sodium thiosulphate solution until only a faint yellow colour remains. Add 10 ml of chloroform to dissolve

the bulks precipitate of bromophenol which, otherwise, is liable to absorb iodine, and follow by a few drops of starch solution. Continue titrating and shaking the contents of the flask until the traces of the blue colour have disappeared. Record the volume of the sodium thiosulphate solution used.

D-2.5 Calculation

Each ml 0.1 N bromide-bromate solution consumed is equivalent to 0.001568 g of phenol

$$\text{Assay, percent by mass} = \frac{6.270\ (50 - f\,v)}{M}$$

Where,

f = factor for the standard sodium thiosulphate solution (*see* **D-2.2.5**);

v = volume in ml, of the standard sodium thiosulphate solution used; and

M = Mass in g, of the sample taken for the test.

D-3 GAS CHROMATOGRAPHIC METHOD (METHOD B)

D-3.1 General

This method determines the concentration of major impurities in trace level, such as mesityl oxide, umene, alpha methylstyrene, 2-methylbenzofuran, acetophenone and dimethylbenzyl alcohol.

D-3.2 Outline of the Method

A known amount of internal standard is added to the sample. A portion of the sample is analyzed by gas chromatography. The concentration of each impurity is calculated relative to the known amount of internal standard that is added. It is useful for individual impurities in the range 10 to 100 mg/kg.

D-3.3 Apparatus

D-3.3.1 *Chromatograph*

Equipped with an on-column injector and flame ionization detector.

D- 3.3.2 *Recorder*

Chemstation or relevant software. With a full scale of 1 mV or µA and a response time of 1 s or less. (in µs)

D-3.3.3 *Integrating Device*

Any device capable of integrating chromatograph peak areas with a repeatability of ±1 percent or less.

D-3.3.4 *Chromatographic Column*

Fused silica, film thickness 0.5 um, length 50 m, inside diameter 0.32 mm or equivalent.

D-3.4 Reagent and Materials

D-3.4.1 *Cross-linked*

Polyethethylene glycol-acid modified

D-3.4.2 *Stationary Phase*

Fatty Acid Phase (FFAP): terephthalic acid modified polyethylene glycols.

D-3.4.3 *Internal Standard*

Sec-butanol

D-3.4.4 *Pure Components for Calibration*

Mesityl oxide, cumene, alpha methylstyrene, 2-methylbenzofuran, hydroxyacetone, acetophenone and dimethylbenzyl alcohol. The purity of each component should exceed 99 weight percent. This above blend should be prepared by using 9:1 pure phenol (99.99 percent) and methanol AR grade.

D-3.4.5 *Phenol*

High purity phenol

D-3.4.6 *Carrier Gas*

Helium/nitrogen/hydrogen

D-3.4.6.1 *Precautions to use hydrogen*

Adequate ventilation must be provided including hydrogen detector and flame proof lighting in storage area to prevent formation of combustible air-hydrogen mixtures.

a) Hydrogen gas cylinders should be kept outside the laboratory in a covered shed to protect from sunlight and rain;

b) All hydrogen gas line couplings should be regularly checked for zero leakage;

c) Most important is that before turning hydrogen gas on, first ensure that the column is fitted inside the GC oven and all connections are leak free, otherwise hydrogen leak concentration can build up and explosions will happen due to high temperature inside the oven;

d) Before removing the GC column for any reason, ensure that hydrogen supply has been turned off; and

e) Any deviation of above will lead to explosion.

D-3.5 Column Condition

Install the column into the chromatograph and precondition it at the elevated temperature described in **D-3.5.1** through **D-3.5.2**. Don't connect the column to the detector block until **D-3.5.2** has been completed.

D-3.5.1 Start the flow of carrier gas and allow the column to age 30 min with no heat.

D-3.5.2 Increase the column temperature to 230°C at the rate of 2 °C/min and hold that temperature for at least 24 h. However, at each 4 h the column should be cooled and restart the heating cycle as mention above till 24 h and connect the column to the detector block after this step been completed.

D-3.5.3 Establish the conditions shown in Table 2 and make repetitive phenol injections until two or more injections exhibit the same peak configuration.

D-3.6 Preparation and Calibration of Standards

D-3.6.1 Use high purity phenol in preparing the calibration standards. Determine the residual impurities in the phenol by the procedure outlined in **D-3.7.**

D-3.6.1.1 *Sample preparation*

The sample must be handled in a molten state at 50 °C. Higher temperature will degrade the sample.

Phenol will freeze at room temperature. The sample and syringe must be kept warm to prevent freezing. An alternative is to add about 10 percent by weight of a solvent, such as a methanol that will not be interference in the chromatography.

D-3.6.2 Prepare a calibration mixture of phenol containing mesityl oxide (MO), cumene, alpha methylstyrene (AMS), 2-methylbenzofuran (2MBF), hydroxy acetone (HA) acetophenone (AP) and dimethylbenzyl alcohol (DMBA). All the above impurity levels should be near the anticipated levels in the phenol sample. If any residual impurity elutes with a known impurity deduct the residual area obtained in **D-3.6.1** (adjusted to sample size) from the area of the impurity.

D-3.6.3 *Standardization Procedure*

Determine the relative response factors (RRF) of each impurity by adding known quantities of impurities and internal standard. Prepare standards with impurity levels that bracket the dynamic range of interest. Use the procedure in **D-3.7** for standardization. Determine the relative response factor, F, as follows,

$$F = \left[W_c \times \frac{A_1}{(A_c - A_b)} \right] \times W_1$$

Where,

W_c = weight of impurity, g;

W_1 = weight of internal standard, g;

A_1 = peak area of internal standard;

A_c = peak area of impurity in calibration blend; and

A_b = peak area of impurity in phenol base stock.

Set the system sensitivity so that all impurity peaks are recorded at adequate levels for data acquisition. Normally, the minimum peak height will be twice that of the baseline noise.

Table 2 Typical or Suggestive Conditions for Chromatographic Separation

(*Clauses* D-3.5.3 *and* D-3.7.1)

Column temperature	240°C
Injector temperature	200°C
Detector temperature	230°C
Carrier gas	Helium/nitrogen/hydrogen
Gas flow rate, ml/min	1.3
Initial temperature °C	110
Initial hold	10
Ramp °C/min	10
Final temperature °C	180
Total run time min	50
Hydrogen flow rate, ml/min	30
Air flow rate, ml/min	300
Specimen size, µl	1

D-3.6.4 *See* Table 3 for typical response factors and retention times. It may vary depends on the nature of GC analysis.

D-3.7 Procedure

D-3.7.1 *See* Table 2 for chromatographic conditions.

D-3.7.2 Add an approximate amount of internal standard to molten phenol and mix thoroughly.

D-3.7.3 Inject 1 µl through auto liquid sampler for achieving repeatability and reproducibility of molten phenol specimen. Phenol is not diluted with water because water may cause ghost peaks.

D-3.7.4 Allow approximately 50 min for components to elute from the column.

D-3.7.5 When phenol elutes, rise the column oven temperature to 180°C. The column should remain at 180°C for approximately 1 h. Before another chromatograph run is attempted, stabilize the oven at 110°C for at least 10 min.

D-3.8 Calculation and Report

D-3.8.1 *Calculation*

Determine the concentration of each impurity using the following formula:

$$M_c = (F_c \times A_c \times M_1) / A_1$$

Where,

M_c = concentration of impurity C, mg/kg;

F_c = relative response factor for impurity C *versus* the internal standard;

A_c = area of impurity C;

M_1 = concentration of internal standard mg/kg; and

A_1 = area of internal standard.

GC purity / dry basis of Phenol (wt. %) = 100 – Sum of all known and unknown impurities.

Absolute purity of phenol (wt. %) = 100 – (Water content + Sum of all known and unknown impurities).

D-3.8.2 Report the concentration of each impurity to the nearest milligram per kilogram.

Table 3 Typical Retention or Elution Order

(*Clause* **D-3.6.4**)

Sl No.	Compound	RT
(1)	(2)	(3)
i)	Acetone	5.88
ii)	Sec-butanol	7.23
iii)	Mesityl oxide	10.02
iv)	Cumene	10.91
v)	Hydroxy acetone	14.72
vi)	α-Methyl styrene	15.06
vii)	2-Methyl benzo furan	21.77
viii)	Acetophenone	23.57
ix)	Dimethyl benzyl alcohol	25.74

NOTES:

1 FID is preferred for phenol purity and trace impurities by using capillary column with spilt/spitless injector.

2 The above gas chromatographic conditions are suggestive/ typical. However, any GC with equivalent column may be used, provided standardization / calibration etc. are done after setting up chromatographic conditions for the desired/required resolution.

3 Phenol produced by process other than the cumene process may have impurities that elute with tetradecane. Before using this test method, investigate this by analyzing the phenol without the internal standard. If interference exists, use other internal standards such as durene or sec-butyl alcohol.

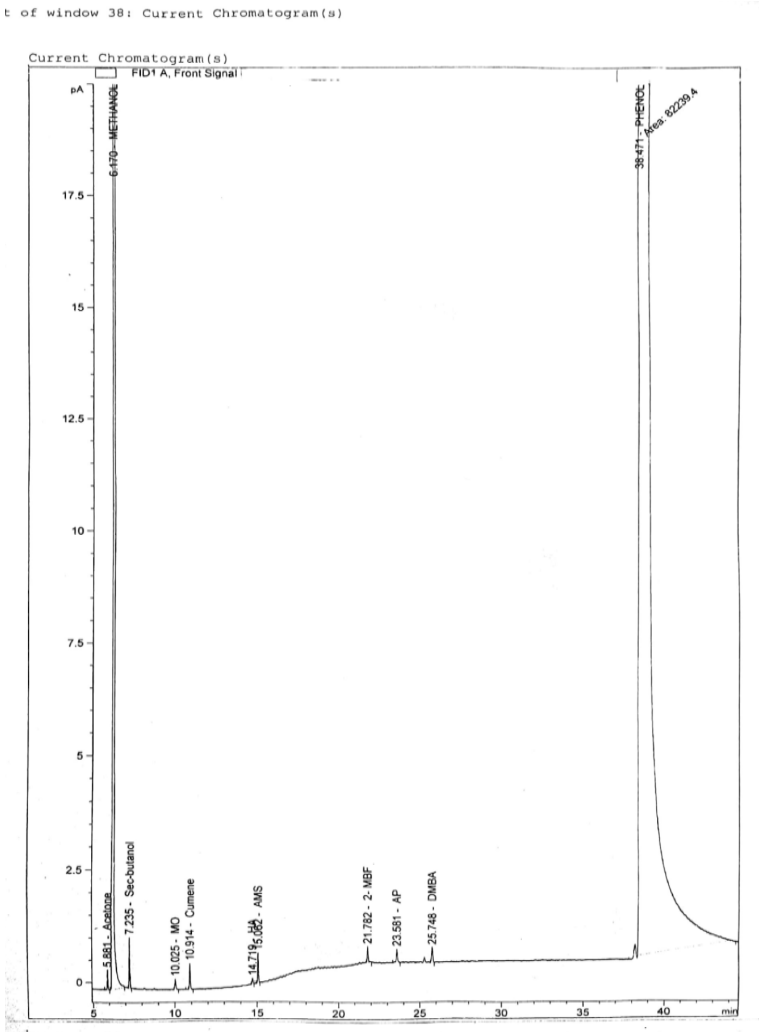

FIG. 3 TYPICAL CHROMATOGRAM AND ELUTION PATTERN

ANNEX E

(*Clause* 6)

SAMPLNG OF PHENOL (CARBOLIC ACID)

E-1 GENERAL REQUIREMENTS OF SAMPLING

E-1.1 In drawing, preparing and handling samples the following precautions shall be observed.

E-1.2 Samples shall not be taken in an exposed place.

E-1.3 The sampling instrument shall be clean and dry when used.

E-1.4 The samples shall be placed in suitable, clean, dry, air-tight, amber or dark blue glass container or any other container, on which the material has no action.

E-1.5 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, the year of manufacture and other important particulars of the consignment.

E-1.6 Sample shall be protected from light.

E-1.7 Phenolic substance burns the skin and may be absorbed into the system through the skin. The sampler shall wear gloves, preferably of polyvinyl chloride and also a face shield. Inhalation of the vapours from hot material shall be avoided.

E-2 SCALE OF SAMPLING

E-2.1 Lot

All the container in a single consignment of material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each shall constitute separate lots.

E-2.1.1 For ascertaining the conformity of the lot to the requirements of the specification, tests shall be carried out for each lot separately.

E-2.2 Sampling from Containers

The number of containers to be selected for sampling shall be in accordance with Table 4.

E-2.2.1 The container shall be selected at random. In order to ensure randomness of selection, random number tables shall be used (*see* IS 4905).

E-3 PREPARATION OF TEST SAMPLES

E-3.1 When the material consists of crystallized masses, the container selected from the consignment shall be allowed to stand in an open tank, on a grid below which a closed steel coil is fitted. When the material is completely melted stir it thoroughly with a clean, dry, smooth, hardwood agitator, and draw samples from each by means of dry glass sampling tube from different depths, care being taken to reduce atmosphere exposure to the minimum.

Table 4 Minimum Number of Containers to be Selected for Sampling from Various Sizes of Lots

(*Clause* E-2.2)

Size of Lot	Size of Gross Sample
Under 25	5
25 to 49	5
50 to 99	10
100 to 199	15
200 to 299	20
300 to 499	30
500 to 799	40
800 to 1299	55
1300 to 3199	75
3200 to 8000	115

E-3.1.1 Out of the material drawn from each individual container, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample sufficient for carrying out triplicate determinations for all the characteristics specified. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

E-3.1.2 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual sample representing the container selected shall be for the purchaser, another for the supplier and the third for the referee.

E-3.1.3 All the individual and composite shall be transferred to separate sample container. These container shall then be sealed air-tight with stoppers and labeled with full identification particulars given in **E-1.5**.

E-3.1.4 The referee test samples, consisting of a composite sample and a set of individual samples, shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the two, to be used in case of any dispute.

E-4 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

E-4.1 The purchaser may examine and test each of the individual samples separately for compliance with the requirements of this standard, or he may decide at any stage of progress of the examination to test a composite sample (*see* **E-3.1.1**) for determining conformity of the lot to this specification.

E-4.2 When the individual samples are separately examined and the results vary from one sample to another, the criteria for judging the quality of the lot for the purpose of acceptance on the results obtained shall be at the discretion of the purchaser, unless otherwise previously agreed to between the purchaser and the supplier.

ANNEX F

(Foreword)

COMMITTEE COMPOSITION

Organic, Chemicals Alcohols and Allied Products Sectional Committee, PCD 09

Organization	*Representative(s)*
Chemical Engineering and Process Development Division, NCL PUNE	Dr C. V. Rode
All India Distilleries Association (AIDA), New Delhi	Shri V. N. Raina
BASF India Limited, Mumbai	Shri Kiran Bhat Shri Hemal Berawala (*Alternate*)
CDRI, Lucknow	Dr Sanjeev Kanojiya
Central Revenues Control Laboratory, Delhi	Dr T. A. Sreenivasa Rao
Chemical and Petrochemicals Manufacturers Association (CPMA), New Delhi	Shri Kamal Nanavaty
Deepak Fertilizer, Pune	Dr Satish Chand Saini Shri Suresh Amle (*Alternate*)
Deepak Phenolics Limited, Bharuch	Dr S. Samal Shri N. R. Sandipkumar Pancha (*Alternate*)
Dow-Corning India Limited, Mumbai	Shri Abraham Barretto. Shri Ritesh Gulabani (*Alternate*)
Indian Chemical Council, Mumbai	Dr Mritunjay Chaubey Shri J. I. Sevak (*Alternate*)
Jubilant life Sciences Ltd, Noida	Shri Hari Mohan Lohani
National Chemical Laboratory, Pune	Dr Udaya Kiran Marelli
UPL Limited, Mumbai	Mr M. D. Vachhani, GM (QA)
Alkyl Amines Chemicals Ltd, Mumbai	Shri S. V. Nikumbhe Shri Sameer Katdare (*Alternate*)
All India Alcohol-Based Industries Development Association (AABIDA), Mumbai	Shri K. L. Raphael Shri Kirti Gajjar (*Alternate*)
Godavari Biorefineries Ltd, Mumbai	Shri Shanul Laxmanrao Pagar Ms Wani A. J. (*Alternate*)
Gujarat Narmada Valley Fertilizers Company Limited, Gujrat	Dr M. J. Kapadia Shri P. R. Desai (*Alternate*)
Hindustan organic chemicals ltd. (HOCL), Mumbai	Shri Deleep Kumar K. Shri V. Mohan (*Alternate*)
India Glycols Limited, Uttarakhand	Shri S. R. Soni Shri Alok Singhal (*Alternate*)
Laxmi Organic Indusrties, Mumbai	Shri J. P. Suryavanshi Dr Vijay S. Mishra (*Alternate*)
Ministry of Chemicals & Fertilizers, New Delhi	Shri O. P. Sharma Shri Varun Singh Poonia (*Alternate*)

Organization	*Representative(s)*
National Test House, Ghaziabad	Shri Debashis Saha Dr Gopal Krishan (*Alternate*)
Reliance India Limited, Mumbai	Shri K. K. Sreeramachandran Shri Vasant Warke
BIS Director General, New Delhi	Shri N. K. Bansal, Scientist 'F' and Head (PCD), [Representing Director General (*Ex-officio*)]

Member Secretary

Shri Chandrakesh Singh
Scientist 'C' (PCD), BIS

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc No.: PCD 09 (15176).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	2323 7617, 2323 3841
Eastern	: 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi, KOLKATA 700054	2337 8499, 2337 8561, 2337 8626, 2337 9120
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg, CHANDIGARH 160019	265 0206, 265 0290
Southern	: C.I.T. Campus, IV Cross Road, CHENNAI 600113	2254 1216, 2254 1442, 2254 2519, 2254 2315
Western	: Manakalaya, E9 MIDC, Marol, Andheri (East), MUMBAI 400093	2832 9295, 2832 7858, 2832 7891, 2832 7892

Branches : AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. COIMBATORE. DEHRADUN. DURGAPUR. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. JAMMU. JAMSHEDPUR. KOCHI. LUCKNOW. NAGPUR. PARWANOO. PATNA. PUNE. RAIPUR. RAJKOT. VISAKHAPATNAM.

Published by BIS, New Delhi